

AD

TECHNICAL REPORT ARCCB-TR-96029

MICROSTRUCTURAL EFFECT ON THE
RESISTIVITY OF THIN SILVER FILMS

S. R. SOSS
B. GITTELMAN
D. TRACY

K. E. MELLO
T.-M. LU
S. L. LEE

OCTOBER 1996



US ARMY ARMAMENT RESEARCH,
DEVELOPMENT AND ENGINEERING CENTER
CLOSE COMBAT ARMAMENTS CENTER
BENÉT LABORATORIES
WATERVLIET, N.Y. 12189-4050



APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

19970123 053

DTIC QUALITY INSPECTED 1

DISCLAIMER

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

The use of trade name(s) and/or manufacturer(s) does not constitute an official indorsement or approval.

DESTRUCTION NOTICE

For classified documents, follow the procedures in DoD 5200.22-M, Industrial Security Manual, Section II-19 or DoD 5200.1-R, Information Security Program Regulation, Chapter IX.

For unclassified, limited documents, destroy by any method that will prevent disclosure of contents or reconstruction of the document.

For unclassified, unlimited documents, destroy when the report is no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE

*Form Approved
OMB No. 0704-0188*

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)			2. REPORT DATE October 1996		3. REPORT TYPE AND DATES COVERED Final		
4. TITLE AND SUBTITLE MICROSTRUCTURAL EFFECT ON THE RESISTIVITY OF THIN SILVER FILMS			5. FUNDING NUMBERS AMCMS No. 6111.01.91A1.1				
6. AUTHOR(S) S.R. Soss (RPI, Troy, NY), B. Gittleman (RPI), D. Tracy (RPI), K.E. Mello (RPI), T.-M. Lu (RPI), and S.L. Lee							
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Benet Laboratories, AMSTA-AR-CCB-O Watervliet, NY 12189-4050			8. PERFORMING ORGANIZATION REPORT NUMBER ARCCB-TR-96029				
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Close Combat Armaments Center Picatinny Arsenal, NJ 07806-5000			10. SPONSORING / MONITORING AGENCY REPORT NUMBER				
11. SUPPLEMENTARY NOTES Submitted to <i>Applied Physics Letters</i> .							
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12b. DISTRIBUTION CODE				
13. ABSTRACT (Maximum 200 words) Thin silver films were deposited using the partially ionized beam deposition technique. It was found that the resistivity of the films is strongly dependent on the incident ion energy used during film growth. The observed resistivity trend exhibits a minimum that is near the bulk value for silver. The x-ray fiber texture analysis technique was used to determine the microstructure of the films. The change in resistivity is shown to be correlated with film microstructure through the change in the fraction of grains whose surface normals are oriented randomly with respect to the substrate normal. The electronic grain boundary reflection coefficient, as extracted from the model of Mayadas and Shatzkes, is shown to also vary with the microstructure.							
14. SUBJECT TERMS Microstructural Effects, Resistivity, Thin Films, Silver, Deposition					15. NUMBER OF PAGES 10		
					16. PRICE CODE		
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED		18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED		19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED		20. LIMITATION OF ABSTRACT UL	

TABLE OF CONTENTS

	<u>Page</u>
ACKNOWLEDGEMENTS	ii
INTRODUCTION	1
EXPERIMENTAL PROCEDURE	1
OBSERVATIONS	2
CONCLUSION	4
REFERENCES	5

TABLES

1. Reflection Coefficient Values Calculated as a Free Parameter Fit of the MS Model to the Experimental Data	6
--	---

LIST OF ILLUSTRATIONS

1. Resistivity of PIB silver films as a function of the ion energy used during deposition	7
2. Resistivity and random volume fraction as a function of the ion energy used in the deposition of the silver films	8

ACKNOWLEDGEMENTS

The authors would like to thank K. Chen for many useful discussions. This work was partially funded by Bausch and Lomb.

INTRODUCTION

Current trends in the microelectronics industry have increased the importance of low resistance interconnect metals. As device structures become smaller and faster, the interconnect signal delay will soon dominate the device performance. Attempts to reduce this delay have been twofold: the reduction of the dielectric constant of the insulating material, and the reduction in the resistivity of the metal interconnect wires. However, there are other problems associated with reduction of interconnect line dimensions. As line widths become smaller, the line current density increases, increasing the risk of electromigration behavior in the film. Thus, the search for a lower resistivity interconnect must be coupled with a means to control the onset of electromigration as well.

Recent work has shown that the orientation of the grains can play a large role in the film properties. Several researchers have been able to show a link between the microstructure and mean time-to-failure rates in thin film conductor lines. This report describes the relationship between the microstructure and the resistivity of thin silver films. Several factors can affect the resistivity of a thin film. For films of high purity, the average grain size is typically considered to be the most important parameter. It is found that a decrease in the volume fraction of grains oriented with a random distribution correlates well with a decrease in the film resistivity.

EXPERIMENTAL PROCEDURE

Using the partially ionized beam (PIB) deposition technique, thin silver films were deposited on glass substrates. The PIB technique has been described in a previous paper (ref 1), and has been shown to be capable of controlling the grain orientation in thin films (refs 2,3). In essence, the PIB utilizes ions derived from the vapor stream (self-ions) to increase the energy at the growth front of the deposited film. The percentage of ions striking the film surface in this fashion can be varied between 0.1 and 5 percent of the neutral flux, and the energy of the incident ions can be varied between 0 and 3.5 KeV. In this study, the glass substrates were held at room temperature during the deposition. To prevent charging of the substrates, an oscillating bias voltage of 1 KHz was applied to the substrate, allowing stray electrons to strike the growth front (ref 4). A deposition rate of approximately 3 Å/s was used. The thickness of the films was approximately 2000 Å. This thickness was chosen as it is well above the mean free path of an electron in bulk silver (540 Å) (ref 5).

Resistivity measurements were performed using a standard four-point probe technique on the metal films deposited on glass. Thickness measurements via a Tencor Alpha Step were used to convert the sheet resistance measurements to resistivity. The circular points in Figure 1 show the variation of the resistivity of the silver films as a function of ion energy used during the deposition of the films.

The solid line in Figure 1 is a plot of the resistivity using the model of Mayadas and Shatzkes (MS) (ref 6). The MS model relates the resistivity in a thin film with the single-crystalline resistivity by the equation

$$\frac{\rho_g}{\rho_s} = 1 - \frac{3}{2}\alpha + 3\alpha^2 - 3\alpha \ln(1 + \frac{1}{\alpha}) \quad (1)$$

where ρ_g is the grain boundary resistivity, ρ_s is the single-crystalline resistivity of the material, and α is the resistivity parameter given by

$$\alpha = \frac{l}{d} \frac{R}{1 - R} \quad (2)$$

where l is the intrinsic electronic mean free path, d is the average grain size in the film, and R is the grain boundary reflection coefficient, which is usually considered to be a constant.

One of the benefits of using the PIB technique is that the orientation of the films can be changed, while keeping the grain size relatively constant. The average grain size of the silver films was determined by measuring the broadening of the x-ray 2θ signal. The grain sizes of the films were found to be between 920 and 1200 Å, with the exception of the 1.3 KeV deposition, which had an average grain size of about 400 Å. The difference in the grain size for this film could be due to the larger deposition rate, 28 Å/s, used.

OBSERVATIONS

For PIB copper films, it has been shown, using secondary ion mass spectroscopy, that oxygen is the only significant impurity (refs 7,8). Oxygen is known to increase the resistivity of metals about $1 \mu\Omega\cdot\text{cm}$ per atomic percent (ref 9). The oxygen content in the PIB copper films was found to be about 5.9×10^{-3} atomic percent near the optimum ion energy. Recent modeling of the PIB process has shown that silver and copper should behave similarly in our system (ref 10).

Using Eq. (1) and inserting the average grain size values and electronic mean free path (540 Å) for the silver films, a theoretical value for the resistivity due to the grain size was found. The reflection coefficient used in the calculation was that for copper, $R = 0.24$ (ref 6). The reason for this choice was due to the lack of data in the literature for a silver reflection coefficient. Differences in this value may shift the calculations, however the general line shape will not change. Adding to this approximately $0.006 \mu\Omega\cdot\text{cm}$ for impurities, an expected value for the resistivity can be calculated, represented by the solid line in Figure 1. The increase in the resistivity in the calculation at an ion energy of 1.3 KeV follows that of experiment due to the smaller grain size. Even should the impurity concentration in the silver films be an order of magnitude higher than that found in copper, this would still mean an increase in resistivity of approximately $0.01 \mu\Omega\cdot\text{cm}$, a change which is an order of magnitude smaller than evidenced in the

experimental data. As can be seen, impurity and grain size effects cannot account for the existence of a minima in the resistivity.

The resistivity effect may be explained by examining the microstructure in the films. The microstructure of the films was examined using a subset of the x-ray pole figure technique, the fiber texture plot. A Scintag XDS2000 operated in the Schultz reflection geometry was used. From the fiber texture data, it was possible to determine the volume fraction of grains whose $\langle 111 \rangle$ planes were oriented with the substrate surface, as well as the volume fraction of grains oriented in a purely random fashion. The $\langle 200 \rangle$ volume fraction could not be measured due to the low x-ray intensity of this reflection in the films. Thus, in our films, the random volume fraction was the complement of the $\langle 111 \rangle$ volume fraction. Figure 2 plots the resistivity and random volume fraction as a function of the ion energy. While the volume of the $\langle 111 \rangle$ orientation changes between films, the width of the distribution as given by the angle at which intensity has fallen to within 10 percent of the peak, ω_{90} , remains relatively constant; ω_{90} varies by less than 10 percent, the average being 21° . As can be seen, there is a very strong correlation between the decrease in random volume fraction and the decrease in resistivity, the minima of both quantities occurring at the same energy. Thus, it appears that the PIB technique is able to produce very low resistivity by affecting the film microstructure via the bombardment of ions during the growth phase of the film.

It is possible to explain this change in the resistivity by examining the theoretical underpinnings of the MS model. The original model makes the assumption that all of the grains in the films are oriented such that the grain boundary normals lie in the film plane. As is seen by the texture analysis of our films, this is not, in general, true. Only those films with very high texture, or very low random volume fractions, will approach the ideal of Mayadas and Shatzkes. Table 1 is a tabulation of the reflection coefficients made by fitting the MS model to the data with R as a free parameter. Parmigiani et al. (ref 11) give evidence that R is not constant; however in their study, the films contain significant void fractions and the more subtle effects of the microstructure, such as texture, which are relevant to films of industrial quality, are masked.

It is apparent from the table that the lowest value of the reflection coefficient coincides with the lowest random volume fraction. The random volume fraction is a measure of grains that are found to be in a purely random distribution in the film. Thus, a decrease in this value indicates there is a decrease in the likelihood that two neighboring grains will have uncorrelated orientations. Because of this, if the grain-to-grain misorientation angle were averaged throughout the film, the result would be lower for those films with a smaller random volume fraction. Calling the average of the grain-grain misorientation angle the average grain tilt, the average reflection coefficient is seen to increase with the tilt of the grain boundary from perpendicular. If the grain boundary potential is assumed to be a delta function potential (i.e., $V(x) = S\delta(x - x_n)$ for the n^{th} boundary), the potential strength, S , can be related to the reflection coefficient by

$$\frac{m}{h^3 d} \frac{S^3}{k_F} 2\tau = \frac{l}{d} \frac{R}{1 - R} \quad (3)$$

where m is the electron mass, k_F is the magnitude of the Fermi wave vector, and τ is the relaxation time constant for scattering from point defects and phonons (ref 6). Using this relation, it can be argued that grain boundary tilt increases the relative strength of the boundary potential, S . This makes good intuitive sense in that as the grain boundaries become more randomly oriented with respect to one another, the lattice distortion between neighboring grains should also increase. This increase in lattice distortion becomes evident in the potential strength parameter, S , which in turn causes an increase in the grain boundary reflection coefficient, R .

CONCLUSION

In conclusion we have shown that the PIB technique can be used to grow thin silver films with bulk-like resistivities at room temperatures. We believe that the reason for the improved resistivity is that the ion-assisted deposition changes the microstructure of the films towards a highly oriented grain texture. By changing the grain orientation in this way, the electronic reflection coefficient at the grain boundaries is lowered, and, hence, the resistivity is improved.

REFERENCES

1. S.-N. Mei and T.-M. Lu, *J. Vac. Sci. Technol. A*, Vol. 6, 1988, p. 9.
2. P. Bai, G.-R. Yang, and T.-M. Lu, *Appl. Phys. Lett.*, Vol. 56, 1990, p. 198.
3. A.S. Yapsir, L. You, T.-M. Lu, and M. Madden, *J. Mater. Res.*, Vol. 4, 1989, p. 343.
4. S.R. Soss, C. Cook, and T.-M. Lu, *Appl. Phys. Lett.*, to be published.
5. N.W. Ashcroft and N.D. Mermin, *Solid State Physics*, Saunders College, 1976.
6. A.F. Mayadas and M. Shatzkes, *Phys. Rev. B*, Vol. 1(4), 1970, p. 1382.
7. P. Bai, G.-R. Yang, L. You, and T.-M. Lu, *J. Appl. Phys.*, Vol. 66, 1989, p. 4519.
8. P. Bai, G.-R. Yang, and T.-M. Lu, *J. Appl. Phys.*, Vol. 68, 1990, p. 3619.
9. C. Kittel, *Introduction to Solid State Physics*, Fifth Edition, John Wiley and Sons, 1976.
10. B. Gittleman, "The Effects of Low Energy Ion Bombardment on the Growth of Thin Films: Model and Experiment," PhD Thesis, Rensselaer Polytechnic Institute, Troy, NY, 1994.
11. F. Parmigiani, E. Kay, T.C. Huang, J. Perrin, M. Jurich, and J.D. Swalen, *Phys. Rev. B*, Vol. 33, 1986, p. 879.

Table 1. Reflection Coefficient Values Calculated as a Free Parameter Fit of the MS Model to the Experimental Data

Ion Energy (eV)	Random Volume Fraction	Reflection Coefficient
1300	0.95	0.296
1450	0.85	0.196
1480	0.79	0.071
1500	0.82	0.175

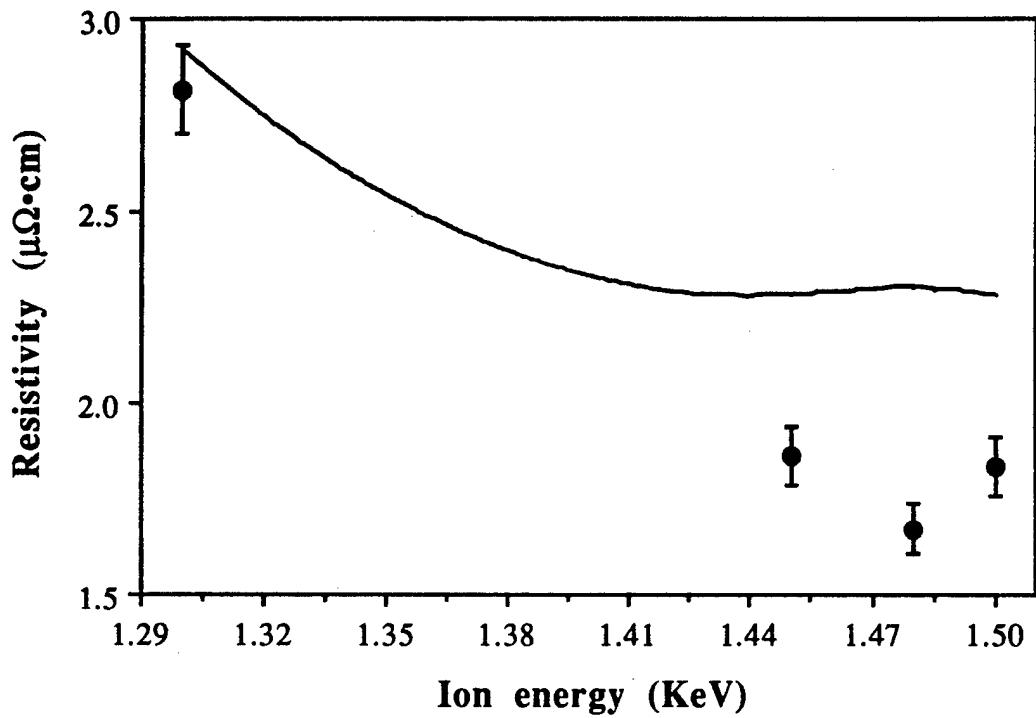


Figure 1. Resistivity of PIB silver films as a function of the ion energy used during deposition. The solid line is the theoretical prediction of the resistivity using the model of Mayadas and Shatzkes and correcting for impurities.

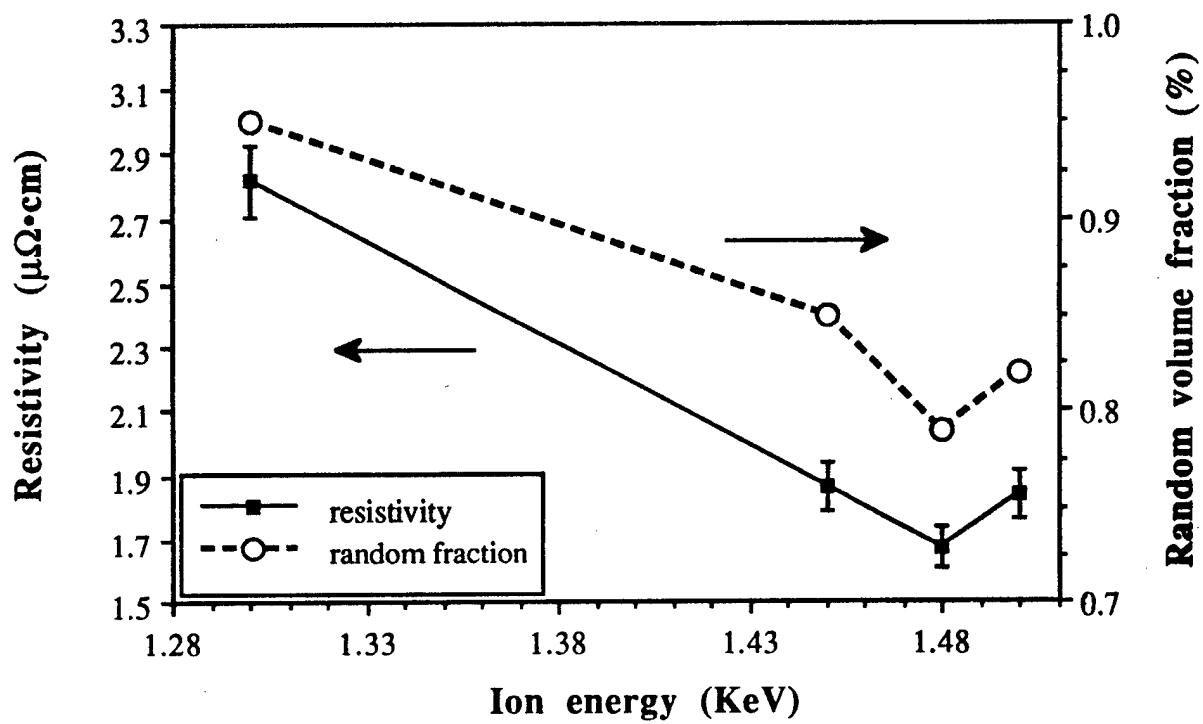


Figure 2. Resistivity and random volume fraction as a function of the ion energy used in the deposition of the silver films. Note the correlation in the minima of both curves.

TECHNICAL REPORT INTERNAL DISTRIBUTION LIST

	<u>NO. OF COPIES</u>
CHIEF, DEVELOPMENT ENGINEERING DIVISION ATTN: AMSTA-AR-CCB-DA	1
-DB	1
-DC	1
-DD	1
-DE	1
CHIEF, ENGINEERING DIVISION ATTN: AMSTA-AR-CCB-E	1
-EA	1
-EB	1
-EC	1
CHIEF, TECHNOLOGY DIVISION ATTN: AMSTA-AR-CCB-T	2
-TA	1
-TB	1
-TC	1
TECHNICAL LIBRARY ATTN: AMSTA-AR-CCB-O	5
TECHNICAL PUBLICATIONS & EDITING SECTION ATTN: AMSTA-AR-CCB-O	3
OPERATIONS DIRECTORATE ATTN: SIOWV-ODP-P	1
DIRECTOR, PROCUREMENT & CONTRACTING DIRECTORATE ATTN: SIOWV-PP	1
DIRECTOR, PRODUCT ASSURANCE & TEST DIRECTORATE ATTN: SIOWV-QA	1

NOTE: PLEASE NOTIFY DIRECTOR, BENÉT LABORATORIES, ATTN: AMSTA-AR-CCB-O OF ADDRESS CHANGES.

TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST

<u>NO. OF COPIES</u>	<u>NO. OF COPIES</u>		
ASST SEC OF THE ARMY RESEARCH AND DEVELOPMENT ATTN: DEPT FOR SCI AND TECH THE PENTAGON WASHINGTON, D.C. 20310-0103	1	COMMANDER ROCK ISLAND ARSENAL ATTN: SMCRI-SEM ROCK ISLAND, IL 61299-5001	1
DEFENSE TECHNICAL INFO CENTER ATTN: DTIC-OCP (ACQUISITIONS) 8725 JOHN J. KINGMAN ROAD STE 0944 FT. BELVOIR, VA 22060-6218	2	MIAC/CINDAS PURDUE UNIVERSITY 2595 YEAGER ROAD WEST LAFAYETTE, IN 47906-1398	1
COMMANDER U.S. ARMY ARDEC ATTN: AMSTA-AR-AEE, BLDG. 3022 AMSTA-AR-AES, BLDG. 321 AMSTA-AR-AET-O, BLDG. 183 AMSTA-AR-FSA, BLDG. 354 AMSTA-AR-FSM-E AMSTA-AR-FSS-D, BLDG. 94 AMSTA-AR-IMC, BLDG. 59 PICATINNY ARSENAL, NJ 07806-5000	1 1 1 1 1 1 2	COMMANDER U.S. ARMY TANK-AUTMV R&D COMMAND ATTN: AMSTA-DDL (TECH LIBRARY) WARREN, MI 48397-5000	1
DIRECTOR U.S. ARMY RESEARCH LABORATORY ATTN: AMSRL-DD-T, BLDG. 305 ABERDEEN PROVING GROUND, MD 21005-5066	1	COMMANDER U.S. MILITARY ACADEMY ATTN: DEPARTMENT OF MECHANICS WEST POINT, NY 10966-1792	1
DIRECTOR U.S. ARMY RESEARCH LABORATORY ATTN: AMSRL-WT-PD (DR. B. BURNS) ABERDEEN PROVING GROUND, MD 21005-5066	1	U.S. ARMY MISSILE COMMAND REDSTONE SCIENTIFIC INFO CENTER ATTN: AMSMI-RD-CS-R/DOCUMENTS BLDG. 4484 REDSTONE ARSENAL, AL 35898-5241	2
DIRECTOR U.S. MATERIEL SYSTEMS ANALYSIS ACTV ATTN: AMXSY-MP ABERDEEN PROVING GROUND, MD 21005-5071	1	COMMANDER U.S. ARMY FOREIGN SCI & TECH CENTER ATTN: DRXST-SD 220 7TH STREET, N.E. CHARLOTTESVILLE, VA 22901	1
		COMMANDER U.S. ARMY LABCOM, ISA ATTN: SLCIS-IM-TL 2800 POWER MILL ROAD ADELPHI, MD 20783-1145	1

NOTE: PLEASE NOTIFY COMMANDER, ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER,
BENÉT LABORATORIES, CCAC, U.S. ARMY TANK-AUTOMOTIVE AND ARMAMENTS COMMAND,
AMSTA-AR-CCB-O, WATERVLIET, NY 12189-4050 OF ADDRESS CHANGES.

TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST (CONT'D)

<u>NO. OF</u> <u>COPIES</u>		<u>NO. OF</u> <u>COPIES</u>
1	COMMANDER U.S. ARMY RESEARCH OFFICE ATTN: CHIEF, IPO P.O. BOX 12211 RESEARCH TRIANGLE PARK, NC 27709-2211	WRIGHT LABORATORY ARMAMENT DIRECTORATE ATTN: WL/MNM EGLIN AFB, FL 32542-6810
1	DIRECTOR U.S. NAVAL RESEARCH LABORATORY ATTN: MATERIALS SCI & TECH DIV WASHINGTON, D.C. 20375	WRIGHT LABORATORY ARMAMENT DIRECTORATE ATTN: WL/MNMF EGLIN AFB, FL 32542-6810

NOTE: PLEASE NOTIFY COMMANDER, ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER,
BENÉT LABORATORIES, CCAC, U.S. ARMY TANK-AUTOMOTIVE AND ARMAMENTS COMMAND,
AMSTA-AR-CCB-O, WATERVLIET, NY 12189-4050 OF ADDRESS CHANGES.

DEPARTMENT OF THE ARMY
ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
BENÉT LABORATORIES, CCAC
US ARMY TANK-AUTOMOTIVE AND ARMAMENTS COMMAND
WATERVLIET, N.Y. 12189-4050

OFFICIAL BUSINESS
AMSTA-AR-CCB-O
TECHNICAL LIBRARY

DEPARTMENT OF THE ARMY

OFFICIAL BUSINESS

Edition of Oct 74 will be used until exhausted.
DA Label 18-1, Sep 83

Defense Technical Information Center
Attn: DTIC-OCP (Acquisitions)
8725 John J. Kingman Road
Ste 0944
Ft. Belvoir, VA 22060-6218